NURUL ASHIKIN BINTI MOHYIDIN MASTER OF SCIENCE 2016

MOLECULARLY IMPRINTED POLYMER AS A SOLID PHASE EXTRACTION SORBENT MATERIAL FOR ATRAZINE CLEAN-UP

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NURUL ASHIKIN BINTI MOHYIDIN

Thesis Submitted in Fulfilment of the Requirement for the Degree of Master of Science in the School of Marine and Environmental Sciences
Universiti Malaysia Terengganu
May 2016

Dedicated to

My beloved parents

Mohyidin Mohd Husin and Sarmini Sapawi

My siblings

Mohd Faiz Mohyidin

Muhammad Norhelmi Mohyidin

Muhammad Naim Mohyidin

Muhammad Arif Mohyidin

Muhammad Najib Mohyidin

Muhammad Afiq Mohyidin

My sister in laws

Nurul Huda Bajuri

Misbahatul Munawarrah Ridzoni

My Niece

Nurul Fatihah Mohd Faiz

Abstract of thesis presented to the Senate of Universiti Malaysia Terengganu
in fulfilment of the requirements for the degree of Master of Science

MOLECULARLY IMPRINTED POLYMER AS A SOLID PHASE EXTRACTION SORBENT MATERIAL FOR ATRAZINE CLEAN-UP

NURUL ASHIKIN BINTI MOHYIDIN

May 2016

Main Supervisor : Associate Proffesor Marinah Mohd Ariffin, Ph.D.

School : School of Marine and Environmental Sciences

Atrazine is an herbicide used as weeds control and it is harmful to environment and human health. The detection of atrazine exists in environment always interfered by other matrices when using conventional extraction methods. Therefore, molecularly imprinted polymer (MIP) for atrazine had been synthesized in order to achieve good reproducibility and more accurate result. MIP was prepared *via* bulk polymerization technique with atrazine as a template. Methacrylic acid (MAA) and ethylene glycol methacrylate acid (EGDMA) were used as monomers and crosslinker respectively. In order to increase pores size of sorbent, acetonitrile with toluene was used as porogen and while azobisisobutyronitrile (AIBN) was used as initiator. NIP was prepared following the same methodology as of with MIP but no addition of template. The polymers were then ground and sieved in the range size of 25 – 38 µm and used as sorbent material for solid phase extraction (SPE).

The physical characterizations were done by using Scanning Electron Microscopy (SEM), Fourier Transform Infrared (FTIR) and Brunauer Emmett Teller (BET).

While, the quantitative analysis was conducted by using High Performance Liquid Chromatography and chromatographic separation using 250 mm x 4.6 mm ID column. Molecularly Imprinted Polymer Solid Phase Extraction (MISPE) method had been optimized to give higher selectivity and good result. The limit of detection (LOD) and limit of quantification (LOQ) for atrazine were 0.02 ppm and 0.07 ppm respectively with correlation coefficient of 0.9997. The recovery values of analytes obtained for MISPE was 64.96% while for Non Imprinted Solid Phase Extraction (NISPE) was 43.89% respectively. This result shows that MISPE is more selective than NISPE towards atrazine due to the presence of imprinted sites in MISPE.

This research was successfully applied in soil samples collected at Marang and Manir. From the analysis of soil samples by MISPE and analyzed by High Performance Liquid Chromatography Ultraviolet Visible (HPLC-UV), concentration of atrazine detected is below than limit detection. All the samples are free from atrazine contamination.

Abstrak tesis yang dikemukakan kepada Senat Universiti Malaysia Terengganu sebagai memenuhi keperluan untuk Ijazah Sarjana Sains

POLIMER MOLEKUL TERCETAK SEBAGAI BAHAN PENJERAP BAGI PENGEKSTRAKAN FASA PEPEJAL UNTUK PEMBERSIHAN ATRAZIN

NURUL ASHIKIN BINTI MOHYIDIN

Mei 2016

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Pusat Pengajian : Pusat Pengajian Sains Marin dan Sekitaran

Atrazin ialah racun rumpai yang digunakan sebagai kawalan rumpai. Ianya berbahaya kepada alam sekitar dan kesihatan manusia. Namun begitu, pengesanan kewujudan atrazine di dalam alam sekitar sering terganggu oleh bahan matriks lain apabila menggunakan kaedah pengektrakan konvensional. Maka polimer pencetakan molekul (MIP) telah disediakan bagi mencapai kebolehdapatan yang baik dan keputusan yang lebih tepat. MIP disediakan melalui teknik pempolimeran pukal dengan menggunakan atrazin sebagai templat. Metil asid akrilik dan etilena glikol dimethacrylate masing-masing telah digunakan sebagai monomer berfungsi dan pemaut silang. Bagi meningkatkan saiz liang MIP, asetonitril dan metilbenzena digunakan sebagai porogen manakala azobisisobutironitril (AIBN) digunakan sebagai pemula. Polimer bukan pencetakan molekul (NIP) disediakan seperti cara penyediaan MIP tanpa kehadiran templat. Polimer yang terhasil dikisar dan ditapis bagi mendapatkan polimer bersaiz diantara 25 – 38 µm dan digunakan sebagai bahan penjerap untuk pengekstrakan fasa pepejal.

Pencirian fizikal telah dilakukan dengan menggunakan Mikroskop Imbasan Elektron (SEM), Spektroskopi Infra Merah Transformasi Fourier (FTIR) dan Brunauer Emmett Teller (BET). Manakala analisis kuantitatif telah dijalankan menggunakan Kromatografi Cecair Berprestasi Tinggi (HPLC) dan pemisahan kromatografi dilakukan dengan menggunakan turus c18 (250 mm x 4.6 mm ID). Kaedah Polimer Molekul Tercetak Sebagai Bahan Penjerap Bagi Pengekstrakan Fasa Pepejal **(**MISPE) telah dioptimumkan bagi mendapatkan pemilihan yang tinggi dan keputusan yang tepat. Had pengesanan dan had kuantifikasi bagi atrazine masing-masing ialah 0.02 ppm dan 0.07 ppm dengan lengkung penentuukur bernilai 0.9997. Peratus perolehan atrazin bagi MISPE ialah 64.96% manakala bagi Tanpa Molekul Tercetak Sebagai Bahan Penjerap Bagi Pengekstrakan Fasa Pepejal (NISPE) ialah 43.89%. Melalui keputusan yang didapati menunjukkan MISPE lebih memilih dari NISPE bagi menentukan atrazin disebabkan kehadiran tapak pencetakan pada MISPE.

Kajian ini telah berjaya dijalankan bagi sampel tanah yang diambil dari Marang dan Manir. Melalui pengekstrakan sampel tanah oleh MISPE dan analisis oleh HPLC-UV, kepekatan atrazin yang dikesan adalah dibawah had pengesanan. Semua sampel didapati bebas daripada pencemaran atrazin.